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Study of the Aging Behavior of Rosin-Alum Sized Papers by Analysis of Mechanical Strength, Optical Properties, and Chemical Composition Following Accelerated Aging

ABSTRACT

Librarians and archivists, as well as book and paper conservators, note that paper produced in the 1700s or even earlier seems to be in much better condition than the paper made from about 1850 to 1950. Researchers have studied this phenomenon by measuring the pH of paper extracts via the TAPPI cold extraction method. This method entails maceration of one gram of paper in cold distilled water (70 ml, 25±5°C), carbon-dioxide-free air or nitrogen gas sparging, and finally pH measurement. In the 1960's, the W. J. Barrow Research Laboratory found a strong correlation between the extract pH of paper and the loss of paper strength, and other researchers have subsequently confirmed this correlation. In order to understand the reason for this aging, we need to briefly shed light on the history of paper sizing methods from the technologist's perspective.

A paper composed of just cellulosic fibers will be water-absorbent because of the hydrophilic nature of cellulose and the porous structure of paper into which water can be readily absorbed. Manufacturers size paper to impart hydrophobicity on the surface of cellulose and/or to reduce the pore structure in order to hold out ink for printing and writing.

Initially papermakers added flour and/or starch to the pulp for internal sizing. The Chinese had used starch for sizing from A.D.768 to the 14th century when they substituted animal glue. Surface sizing with gelatin and potassium aluminum sulfate was introduced in the 13th century. Zinc sulfate occasionally replaced potassium aluminum sulfate in gelatin sizing and this combination lasted until Mortiz Illig's invention of rosin-alum sizing in 1807. The rosin was added to the pulp and precipitated with the aluminum salt to size the paper internally. Papermakers primarily used this combination until the 1980s when new neutral or alkaline papermaking processes became prominent. The common alkaline sizing agents today are alkylketene dimer (AKD) and alkenylsuccinic anhydride (ASA).

From the history of paper sizing methods, we know that the paper made from 1850 to 1950 was internally sized with rosin and papermaker's alum (aluminum sulfate) at an acidic pH, usually between a pH of 4 and 5. This low pH was required to allow the aluminumIII cations to form bonds between the rosin and cellulose fibers. The presence of these aluminum ions was found to increase the rate of cellulose degradation, which embrittles the paper during long-term storage.

Most paper research requires some form of aging. The technique of accelerated aging to observe the deterioration of paper through heating was perhaps introduced in 1899. This technique was developed and further refined during the 1920s through several tests in the United States and Sweden. An artificial aging method exposing samples for 72 hours at 100°C was considered equivalent to 18–25 years of natural aging. In the 1950s, researchers noted that relative humidity had a strong effect on the chemical reactions causing paper degradation. They also determined that the reactions that cause degradation have differing activation energies. These observations prompted several researchers to advocate using the Arrhenius equation in accelerated aging studies and a relative humidity equivalent to long-term natural aging.

In recent years, some researchers have reported a relationship between the embrittlement of paper and various sizing methods using a pure cellulose paper, normally Whatman filter paper. It is hard to find any articles in the conservation science literature, however, that report the changes in mechanical, chemical, and optical properties of paper that was sized internally with rosin and alum.

As part of the Heritage Science for Conservation project at The Sheridan Libraries, Johns Hopkins University, funded by the Andrew W. Mellon Foundation, special papers were made under TAPPI standard conditions using cotton rag fiber that was sized with rosin and alum. Subsequently, these TAPPI handsheets were subjected to accelerated aging and tested using a variety of instruments common in the paper industry. Further research will involve deacidifying those handsheets,

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again aging the paper, and testing those sheets in a similar manner. The aging conditions selected include variations of time, temperature, and relative humidity. The results of this research will be published at a later date.

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